

HEAT TREATMENT  
OF HIGH SPEED TOOL STEELS

BY

H. PERLSTEIN  
D. B. LESSER

ARMOUR INSTITUTE OF TECHNOLOGY

1914

669.1

P 42



Illinois Institute  
of Technology  
**UNIVERSITY LIBRARIES**

AT 346

Perlstein, H.

Heat treatment of chrome  
tungsten high speed tool

**For Use In Library Only**







A Thesis  
Presented by  
Harris Perlstein  
and  
David Barnes Lesser  
to the  
President and Faculty  
of  
Armour Institute of Technology  
for the degree of  
Bachelor of Science  
Having Completed the Prescribed  
Course in Chemical Engineering  
May 28, 1914.

Approved by

A. M. McCormack  
Professor of Chemical Engineering

H. M. Raymond  
Dean of Engineering Studies

L. C. Morin  
Dean of Cultural Studies

ILLINOIS INSTITUTE OF TECHNOLOGY  
PAUL GALVIN LIBRARY  
35 WEST 33RD STREET  
CHICAGO, IL 60616





663.1  
P42

Heat Treatment of  
Chrome Tungsten High Speed  
Tool Steels.

By  
Harris Perlstein  
and  
David Barnes Lesser.

1 June '14.

24859



# Outline Index of Thesis.

Introduction	Page I.
Chemical Analysis	" 1.
1. Sampling.	
2. Methods of Analysis.	
a. Silica.	
b. Tungsten.	
c. Chromium.	
d. Manganese.	
e. Nickel.	
f. Phosphorus.	
g. Sulphur.	
h. Carbon.	
i. Molybdenum.	
j. Vanadium.	
Heat Treatment	" 13.
1. Specimens.	
2. Apparatus.	
3. Method.	
Metallography	" 15.
1. Polishing.	
a. Apparatus.	
b. Method.	
2. Etching.	



### 3. Microphotography

a. Apparatus.

b. Method.

### 4. Results.

Interpretation of Results and

Conclusions

Page 18.

### Appendix

Table guide to marked specimens.

Table guide to numbered photographic  
plates.



The exact conditions of heat treatment which will insure the maximum of cutting strength and life to a high speed tool are a matter of much interest to the user of the tool and the manufacturer of the tool steel. In spite of the liveness of the topic, so far as is known to the authors little accurate information is to be had concerning it. Experience with the various steels on the market, together with some few researches has pointed out certain temperature ranges for preheating which, coupled with certain methods of quenching, produce tools that work satisfactorily in a lathe. It is to be remembered, however, that a tool may work satisfactorily from the operator's viewpoint and yet be producing, per dollar input to the lathe, less than it could be made to produce by scientific heat treatment. Careful investigation should furnish data for determining what heat treatment is best in any particular case; but it is this data which is available for very few of the tool steels used in this country.

The plan of study originally outlined by the authors was briefly as follows:

Certain of the better known high speed steels were to be worked with in an endeavor to learn what heat treatment would give best results in each case. Specimens of these steels were to be treated at various temperatures and quenched, and the treated specimens studied and compared by means of microphotography and cutting tests in

The first of these is the fact that the

the maximum of output is reached at a

high speed tool, and the interest in the

use of the tool and the number of the tool.

In spite of the interest in the topic, so far as is

known to the author, little accurate information is so

far concerning it. The difference with the various

steel of the market, together with some few

has pointed out certain temperature ranges for present-

ing, coupled with detailed sections of processing, and

these tools have been satisfactorily in a few.

to be remembered, however, that a tool may work satis-

factorily from the operator's viewpoint and yet the pro-

duction, per roller front to the latter, less than is

could be made to produce by rollers of the same size.

Certain investigation should furnish data for deter-

mining what heat treatment is best in any particular

case, but it is this data which is available for very

few of the tool steels used in this country.

The plan of study originally outlined by the author

was originally as follows:

Control of the better known high speed steels were

to be tested with in an attempt to find what heat treat-

ment would give best results in each case. The results

of these steels were to be treated at various temperatures

and the results compared with the results of the other

group of steels of lower speed and lower cost.



a lathe. Analyses of the steels used were to be made to learn the present practice of high speed steel manufacturers, and in the hope that some clew of correlation between chemical composition and behavior of the treated steels might be developed.

When work on the subject was begun it was decided that the mode of quenching to be used would be by cooling in an air blast, since this corresponds to most commercial practice.

In performing the analytical work difficulties were encountered which upset the tentative time schedule laid out at the start. It was found necessary to narrow the limits of the further work and the cutting tests planned on were therefore abandoned.

The work actually accomplished consisted in analysing, heat treating and obtaining microphotographs of specimens of the following chrome tungsten high speed tool steels:

Brand	Manufacturer or Dealer	Price Dollars per pound
Rekord Superior	Kent & Simpson	.95
Poldi	F. A. Frasse & Co.	1.00
Blue Chip	Frith-Sterling Steel Co.	.75
Novo	H. Boker	—
Arc (Jessops)	Crerar Adams & Co.	.75
Bethlehem	L. P. Nyerson	.60

The authors wish to express appreciation for helpful supervision of Professor H. McCormack and the suggestions of Dr. C. A. Tibbals, and Mr. H. B. Pulsifer.

...analysis of the whole and to be able to  
learn the present practice of high speed steel manu-  
facture, and in the hope that some of the correlation  
between chemical composition and behavior of the treated  
steels might be developed.

When work on the subject was so far advanced  
that the mode of quenching to be used would be of con-  
siderable importance, since this corresponds to most  
commercial practices.

In performing the analytical work difficulties were  
encountered which kept the tentative time schedule  
laid out at the start. It was found necessary to narrow  
the limits of the further work and the cutting tests  
planned on were therefore abandoned.  
The work actually completed consisted in ana-  
lyzing, heat treating and determining microhardness of  
specimens of the following steels: (Table 1)  
Steel Specimens:

Steel	Heat Treatment	Microhardness
SAE 52100	Oil	200
SAE 52100	Oil	200
SAE 52100	Oil	200
SAE 52100	Oil	200
SAE 52100	Oil	200
SAE 52100	Oil	200
SAE 52100	Oil	200
SAE 52100	Oil	200
SAE 52100	Oil	200
SAE 52100	Oil	200

The last series of experiments was made for the  
purpose of determining the effect of the heat treat-  
ment on the mechanical properties of the steels.

## Chemical Analysis.

### Sampling.

Samples for analysis were obtained by drilling the annealed high speed tool steels with a tempered carbon steel twist drill. The piece to be drilled was surfaced on the grindstone until free of oxide and paint and the drilling was then accomplished without the use of lubricant of any kind.

All the steels with the exception of Record were received from the manufacturer or dealer in annealed bars. Attempts to anneal pieces of Rekord by heating white hot in a forge and cooling in a lime box, and by heating at various temperatures from 800° C. to 1200° C. in a muffle and allowing to cool in the muffle failed. Success was finally had by packing the steel in lime in an iron bomb, packing this in lime within a second iron bomb, heating to about 1200° C. and allowing the whole to cool off with the furnace.

### Methods.

Below are given the analytical methods used, together with comments on difficulties encountered.

#### Silica.

A two gram sample is decomposed with thirty cc of aqua regia, taken dry on the hot plate and baked for one hour at 110° C. The baked mass is taken up in hydrochloric acid and the insoluble matter filtered off, and washed free from iron with warm dilute hydrochloric (1:2). The tungstic and

Results.

Analysis for analysis was obtained by the following method. The analysis was made on a sample of carbon steel twist drill. The steel was in the form of a bar and was cut into three pieces of equal length. The first piece was used for the analysis of the steel as received. The second piece was used for the analysis of the steel after being heated to a temperature of 1000°C. and then cooled. The third piece was used for the analysis of the steel after being heated to a temperature of 1000°C. and then cooled in oil.

All the steels were analyzed for the presence of carbon, manganese, phosphorus, and sulfur. The results of the analysis are given in Table I. The results show that the steel as received contains 0.25% carbon, 0.60% manganese, 0.015% phosphorus, and 0.010% sulfur. The steel after being heated to 1000°C. and then cooled contains 0.25% carbon, 0.60% manganese, 0.015% phosphorus, and 0.010% sulfur. The steel after being heated to 1000°C. and then cooled in oil contains 0.25% carbon, 0.60% manganese, 0.015% phosphorus, and 0.010% sulfur. The results show that the steel is stable to heat treatment.

Method.

Below are given the analytical methods used for the determination of the elements in the steel. The methods are given in detail in the Appendix.

Carbon.

The carbon in the steel was determined by the Leco method. The steel was cut into a small piece and was weighed. The piece was then placed in a Leco carbon analyzer and the carbon content was determined. The results are given in Table I.



silicic acids remaining on the paper are washed twice with warm water and the tungstic acid dissolved from the paper with warm concentrated ammonia. The paper and residue are ignited in a platinum crucible and weighed. Ten cc of hydrofluoric acid and two drops of sulphuric acid are added and the ignited material is evaporated dry on the hot plate, reignited and reweighed. The loss in weight is recorded as silica.

It may be objected that the treatment with ammonia dissolves some silica in addition to the tungstic acid, but the thorough baking and relatively small quantity of silica present in the samples worked with is believed to obviate this criticism.

#### Tungsten.

One half gram of the sample is decomposed on the hot plate with 20 cc of aqua regia and evaporation continued until the material is just moist. Five cc of nitric acid is added, evaporation on the hot plate continued to syrupy consistency and the beaker transferred to the water bath. The material is taken dry and dehydrated on the water bath for four to five hours. The mass is moistened with nitric acid and warmed on the water bath, enough water is added to dilute the nitric to about 1:3 and the residue is filtered off and washed free of iron with nitric acid (1:3). The filtrate and washings are set aside for use in determining chromium. The material on the filter paper is washed once or twice with hot water and the tungstic acid is then dissolved from the paper with warm ammonium hydroxide. The filtrate is caught in a tared platinum dish and evaporated almost

silicic acid remains on the paper and is left twice  
with warm water and the silicic acid dissolved from  
the paper with warm concentrated ammonia. The paper  
and residue are ignited in a platinum crucible and  
weighed. The loss is of hydrochloric acid and is  
of silicic acid and is added to the limited material  
is evaporated dry on the water bath, residue and re-  
weighed. The loss in weight is recorded as silica.  
It can be objected that the treatment with ammonia  
dissolves some silica in addition to the silicic acid,  
but the thorough baking and relatively low quantity  
of silica present in the samples worked with is believed  
to obviate this criticism.

#### Tested.

One half gram of the sample is weighed on the  
hot plate with 50 cc of water and evaporated con-  
tinued until the material is just moist. Five cc of  
nitric acid is added, evaporation on the water bath con-  
tinued to syrupy consistency and the beaker transferred  
to the water bath. The material is heated and evaporated  
to dryness on the water bath for four to five hours. The mass is  
dissolved with nitric acid and warmed on the water bath.  
Enough water is added to dilute the nitric acid to 100 cc  
and the solution is filtered off a small amount of silica  
with nitric acid (1:1). The filtrate and washings are  
reweighed and are determined as carbonates. The material  
on the filter is washed once or twice with water  
water and the filtrate and washings are added to the  
portion with warm ammonia solution. The filtrate is  
weighed in a tared flask and evaporated silica

dry on the sand bath. Nitric acid is added in slight excess and the evaporation continued to dryness. Subsequent ignition (preferably in a muffle) gives, by gain in weight, the tungstic oxide corresponding to the tungsten content of the steel.

The above method was decided on at the suggestion of Dr. C. A. Tibbals, after the method outlined below had been found to give low results.

Two grams of the sample was treated with aqua regia, the hydrochloric was replaced by nitric and the mass was baked on a hot plate. The baked material was taken up in nitric acid, the insoluble matter filtered off, washed free of iron and dissolved back into the original beaker with ammonium hydroxide. The solution was filtered (to get rid of silica) and the tungsten reprecipitated by acidifying with nitric acid. The precipitate was filtered off, ignited in platinum and weighed as tungstic oxide.

This method was tried out on B. S. chrome-tungsten steel number 31 and gave results several per cent low (approximately 7% low on a steel containing 19% of tungsten). The large error was due to difficulty in reprecipitating the tungstic acid from ammonia solution from a bulk of about 300 to 400 cc. (It was noted here that the precipitate was generally very white, indicating a high degree of hydration). By evaporating the ammonium filtrate almost dry on the water bath and then acidifying, far better

dry on the sand bath. Nitric acid is added in slight  
excess and the vaporization continued to dryness. The  
residue is ignited (preferably in a metal) and the  
gain in weight, the volatile oxide corresponding to the  
loss of the volatile oxide of the metal.  
The above method was devised by Mr. A. T. M. and  
or Mr. A. T. M., after the method outlined in the  
and then found to give for results.  
Two grams of the sample was treated with dilute  
the arsenic trioxide was replaced by nitric acid and the  
the residue on a sand bath. The residue was then  
up in nitric acid, the insoluble matter filtered off,  
various parts of iron and aluminum were added to the original  
beaker with ammonium hydroxide. The solution was then  
added to the rest of the acid and the ammonia was added  
test by adding a few drops of nitric acid. The precipitate was  
filtered off, ignited in a platinum dish and weighed as  
oxide.  
The method was tried out on a sample of arsenic  
and about 1.1 g. gave 0.1 g. of arsenic trioxide. The  
precipitate was then on a sand bath and the residue  
The residue on the sand bath was then ignited, leaving  
the residue and the arsenic trioxide was a black of  
about 0.1 to 0.2 g. It was noted that the precipi-  
tate was normally very white, indicating a high degree  
of hydration. It was noted that the arsenic trioxide  
was dry on the sand bath and the residue was a black



results were obtained by Dr. Tibbals in attempting to check the authors, but the filtrate after the acidification still contained a very appreciable quantity of tungsten, hence the method first given was adopted, the sample being cut from two grams to one half gram. The dehydration is carried out on the water bath rather than the hot plate in order that chromium, which is determined on the same sample be not rendered insoluble.

#### Chromium.

The filtrate from the tungstic and silicic acids is evaporated nearly dry, 20 cc of nitric acid and two grams of potassium chlorate are added and the solution boiled for ten minutes. Ten cc of nitric acid and one gram of potassium chlorate are added and the solution boiled again for ten minutes. The chromium is now completely oxidized to chromate. The solution is diluted somewhat, allowed to cool, and neutralized with ammonium hydroxide. The solution is filtered from the iron hydroxide and washed with warm water. The iron hydroxide is redissolved into the original beaker with warm hydrochloric acid (1:1), reprecipitated and washed, the reprecipitation and washing being continued until the iron precipitate is free of chromium. (Three to four precipitations are necessary), The filtrate and washings are acidified with acetic acid and lead acetate is added in excess. The precipitate is allowed to settle, filtered off and washed a few times with water and dissolved into the original beaker with hydrochloric acid, (1:1)



The solution is made up to about 400 cc, 20 cc of a fifteen per cent potassium iodide solution are added and titration is carried out against tenth or twentieth normal sodium thiosulphate solution with starch as the indicator.

The above method gave satisfactory results and was successfully checked by Dr. Tibbals on the one sample run by him simultaneously with his work on the tungsten.

The solution is made up to about 100 cc, 20 cc of a 10% per cent potassium iodide solution are added and the solution is carried out against tenth or twentieth normal solution of phosphate solution with starch as the indicator. The above method gave satisfactory results and was successfully checked by Dr. Tibbels on the one sample run by him simultaneously with his work on the indicator.

### Manganese.

Manganese was determined by a slight modification of the Ford Williams method (B. S. circular no. 14).

Two grams of steel are decomposed with 30 cc of aqua regia and taken very nearly dry on the hot plate. Nitric acid is added and evaporation continued for a few cc to replace hydrochloric. Nitric acid (sp. gr. 1.2) is added and the insoluble matter filtered off and washed with nitric acid ~~xxxx~~ (1:3). The solution is evaporated to a few cc, 30 cc of concentrated nitric acid and 2 grams of potassium chlorate are added and it is boiled for ten minutes. Ten cc of nitric acid ~~and~~ one gram of potassium chlorate are added and the boiling is continued for ten minutes. The precipitate is filtered on asbestos, washed two or three times with strong nitric acid and then with water. The asbestos pad is transferred to a flask, shaken up with excess standard ferrous sulphate solution until the manganese dioxide is dissolved, and the excess of ferrous sulphate is titrated with potassium permanganate.

### Nickel.

One gram of steel is dissolved in 25 cc of hydrochloric acid (sp. gr. 1.16). The solution is filtered and the residue washed with hot water. One gram of tartaric acid is added to the filtrate which is heated to 50° C. Sufficient dimethylglyoxime solution containing 2.5 grams in 220 cc of 98% alcohol, is



# ANALYSIS

The sample was dissolved in a small amount of water and the solution was transferred to a 100 ml. volumetric flask. The flask was then filled with water to the mark and the solution was mixed thoroughly. A portion of the solution was then transferred to a beaker and the pH was measured. The pH was found to be 1.2. The solution was then titrated with 0.1 N sodium hydroxide solution. The endpoint was reached when the color changed from red to yellow. The volume of sodium hydroxide solution used was 15.0 ml. The concentration of the sample was calculated to be 0.015 N.

## RESULTS

The sample was found to contain 0.015 N of the active ingredient. The results of the titration are shown in the table below. The sample was found to be pure and the results are in good agreement with the theoretical values.

added to precipitate the nickel present. ( 1% nickel requires twenty cc of the glyoxime solution). The solution is made very faintly alkaline with ammonia, the nickel coming down as a scarlet flocculent precipitate, which is filtered on a gooch crucible, washed well with hot water, dried at 100° C. and weighed, the weight obtained being converted to nickel by use of the factor .2031. (NiO<sub>8</sub> H<sub>14</sub> N<sub>4</sub> O<sub>11</sub> X.2031 equals Ni.)

#### Sulphur. ( Evolution Method. )

Five grams of sample are dissolved in 60 cc of hydrochloric acid in a 500 cc erlenmeyer flask fitted with a dropping funnel and connected to an absorption flask containing a 100% solution of sodium hydroxide. The acid is run in quickly from the dropping funnel and the solution is boiled for fifteen minutes. The contents of the absorption flask are transferred to a beaker, diluted to 400 cc, cooled and made acid with hydrochloric. Titration of the hydrogen sulphide present is made against twentieth normal iodine solution.

#### Phosphorus.

Phosphorus is determined alkalimetrically modifying the usual method according to the suggestions of Cain and Tucker ( B. S. Technologic Paper No. 24 ) to avoid errors due to the presence of vanadium. Later it was found by qualitative tests that no vanadium was present in the steels worked with.





Two grams of steel are dissolved in aqua regia and evaporated nearly dry, then taken up in five cc of nitric acid and run nearly dry again to replace hydrochloric. Nitric acid (sp. gr. 1.135) and ten cc of potassium permanganate solution (fifteen grams per l.) are added and the solution boiled. Sodium thiosulphate solution is added to dissolve manganese dioxide and the solution boiled and filtered. The filtrate is cooled to fifteen to twenty degrees C. and five cc of saturated ferrous sulphate solution and two or three drops of concentrated sulphur<sup>ous</sup> acid are added. Forty cc of ammonium molybdate solution are added and the solution shaken for fifteen minutes at about 20° C. The precipitate is filtered and washed with 1% nitric acid, followed by 1% potassium nitrate solution, until the washings are no longer alkaline. The precipitate is then dissolved in excess standard sodium hydroxide solution and the excess hydroxide titrated back with standard nitric acid, using phenolphthalein as indicator. The sodium hydroxide is standardized against prepared ammonium phosphomolybdate.

Total Carbon  
(Direct Combustion)

The electric combustion furnace shown in photograph number one is heated to above 950° C and oxygen is run through the apparatus to displace the air. The oxygen passes thru potassium hydroxide solution and over stick potassium hydroxide and then thru the furnace. The pro-





Photograph No. 1



ducts of combustion pass thru two U tubes containing respectively, granulated zinc and calcium chloride and then thru a Geisler absorption bulb. The liquid and solid potassium hydroxide free the oxygen from carbon dioxide and water. The granulated zinc removes acid and sulphur fumes from the gasses of combustion which are dried by the calcium chloride and give up their carbon dioxide to the 50% potassium hydroxide solution in the Geisler bulb.

Two grams of drillings are placed in an alundum boat which is pushed quickly to the center of the silica tube of the furnace (the latter being <sup>H.T.</sup>), Oxygen is passing at the start at a fairly rapid rate and is controlled so that bubbles are continually passing thru the Geisler bulb during the run. The run is continued for 15 minutes. The increase in weight of the Geisler bulb, which has been previously weighed full of oxygen under atmospheric pressure, and which is again weighed under the same conditions, gives the weight of carbon dioxide corresponding to the carbon content of the sample.

#### Vanadium and molybdenum

(qualitative)

Qualitative tests for vanadium and molybdenum revealed the absence of these elements in all cases. The following method is adapted from that used by Hillebrand to detect vanadium in rocks (Freadwell-Hall Volume 1, Page 489).



direct of combustion from the two combustion  
 respectively, granulated lime and calcium chloride and  
 then form a wetter absorption bulb. The liquid and  
 solid potassium hydroxide free the oxygen from carbon  
 dioxide and water. The granulated lime removes acid  
 and sulphur fumes from the gases of combustion which  
 are dried by the calcium chloride and give up their car-  
 bon dioxide to the potassium hydroxide solution in  
 the wetter bulb.

Two grams of grillings are placed in an aluminum  
 boat which is pushed quickly to the center of the slide  
 tube of the furnace (the latter being <sup>hot</sup> oxygen is  
 passing at the start at a fairly rapid rate and is con-  
 trolled so that bubbles are continually passing thru  
 the gasifier bulb during the run. The run is continued  
 for 15 minutes. The increase in weight of the wetter  
 bulb, which has been previously weighed full of oxygen  
 and atmospheric pressure, and which is again weighed  
 under the same conditions, gives the weight of carbon  
 dioxide corresponding to the carbon content of the sample.

#### Vanadium and Molybdenum

(Qualitative)

Qualitative tests for vanadium and molybdenum re-  
 volved the presence of these elements in all cases. The  
 following method is adapted from that used by  
 to detect vanadium in rocks (see Vol. 1, p. 100).

A sample of about 1 gram of the steel is decomposed with aqua regia, evaporated the small volume, diluted and made alkaline with ammonium hydroxide. The precipitate is filtered off, dried and fused in platinum or porcelain, with 10 grams of sodium carbonate and 2 grams of sodium nitrate. Fusion is leached with hot water and a little alcohol is added to reduce the manganate formed. The solution may contain phosphorous, chromium, vanadium, molybdenum and tungsten. It is nearly neutralized with nitric acid and evaporated nearly dry, taken up in water and filtered. A few cc of mercurous nitrate are added to the filtrate. Mercurous, phosphate, chromate, vanadate, molybdate and tungstate may precipitate. The solution is boiled and filtered and the precipitate is dried, ignited in platinum and fused with a little sodium carbonate. The fusion is leached with water and acidified with sulphuric acid. Hydrogen sulphide is passed. A brown precipitate soluble in ammonium sulphide, and reprecipitated by the addition of acids, is molybdenum trisulphide and proves the presence of molybdenum. Any precipitate caused by the hydrogen sulphide is filtered off and the excess hydrogen sulphide removed by boiling while a stream of carbon dioxide is passed thru the solution. The solution is evaporated to dryness and the excess of sulphuric acid is removed by heating in an air bath. The residue is

A sample of about 1 gram of the steel is dissolved  
with aqua regia, evaporated the small volume, filtered  
and as a filtrate with ammonium hydroxide. The precipi-  
tate is filtered off, dried and fused in platinum or  
porcelain, with 10 grams of sodium carbonate. The mass  
of sodium nitrate. Fusion is repeated with 10 g  
and a little alcohol is added to reduce the weight  
for the solution may contain hydrochloric acid, chromic  
vanadium, molybdenum and tungsten. It is nearly neutral-  
ized with nitric acid and evaporated nearly dry, taken  
up in water and filtered. A few cc of potassium nitrate  
are added to the filtrate. Hydrochloric, phosphoric, chromic,  
vanadic, molybdic and tungstic acids are precipitated. The  
solution is boiled and filtered and the filtrate is  
boiled, ignited in platinum and fused with a little soda  
ash. The residue is dried with water and  
dissolved with dilute acid. Hydrochloric acid is  
passed. A white precipitate soluble in ammonium sul-  
phate, and reprecipitated by the addition of water,  
molybdenum trioxide and gives the residue of  
molybdenum. Any precipitate caused by the hydrogen  
sulphide is filtered off and the molybdenum trioxide  
is removed by boiling with a stream of carbon di-  
oxide as passed thru the solution. The solution is  
evaporated to dryness and the residue of molybdenum trioxide  
removed by heating in an air oven. The residue is



dissolved in the few cc of water and shaken with a few drops of hydrogen peroxide. The appearance of a brownish yellow color shows the presence of vanadium.

a solution in the low of water and shaken with a  
 few drops of hydrochloric acid. The appearance of a  
 brownish yellow color shows the presence of vanadium.

## ANALYTICAL RESULTS

	Rekord	Poldi	Bethlehem	Arc	Blue Chip	Novo
Silica	.18%	.17%	.28%	.13%	.18%	.48%
Manganese	.06	nil	.427	.065	.276	.290
Phosphorus	.01	.029	.038	.018	.052	.033
Tungsten	13.69	17.56	17.88	12.99	15.63	16.82
Chromium	2.35	2.33	2.54	3.90	2.48	1.50
Supphur	.003	.009	.011	.025	.008	.015
Carbon	.56	.58	.52	.46	.70	.60
Nickel	nil	nil	nil	nil	nil	nil
Vanddium	nil	nil	nil	nil	nil	nil
Molybdenum	nil	nil	nil	nil	nil	nil

2007: 2401-2421

## HEAT TREATMENT

### Specimens

The steels were cut into lengths of five inches and numbered by a steel punch (see guide in appendix) to furnish the specimens for heat treatment.

### Apparatus

An electric muffle furnace was available which answered the requirements of uniform temperature throughout the heating space and easy temperature control by the apparatus. This was used for the heat treatment. It was a Hoskins electric furnace type F. C. shown in photograph No. 2. This furnace was set well up from the floor and well heat insulated. The heating occurs from two sides and the top. The control switch board is shown a short distance back from the furnace.

It has been mentioned that cooling in an air blast was decided on as the mode of quenching to be employed. Pipe was run out from an air line to about three feet in front of the furnace, then down to the floor, and finally about 20 inches of 3-inch pipe was connected up vertically to serve as a cooling chamber. The arrangement is shown in photograph No. 3, which also shows how it was placed relative to the furnace. As the air entered the cooling chamber from the bottom thru a half-inch pipe it was not thought advisable to allow the specimen to rest on the bottom of the cooling chamber. Such a course would probably have

Experiment

The steels were cut into lengths of five inches and numbered by a steel punch (see table in appendix) to furnish the specimens for heat treatment.

Apparatus

An electric oil fire furnace was available which answered the requirements of uniform temperature throughout the heating space and easy temperature control by the operator. This was used for the heat treatment. It was a box-like electric furnace type, as shown in photograph No. 1. The furnace was set well up from the floor and well vented to the top. The heating occurs in two sides and the top. The control switch is about a short distance back from the furnace.

It has been mentioned that cooling in an air blast was desired on all the rods of specimens to be compared. This was run out from an air line to about three feet in front of the furnace, then down to the floor, and finally about 30 inches of 1/2-inch pipe was connected up vertically to serve as a cooling chamber. The arrangement is shown in photograph No. 2, which also shows how it was placed relative to the furnace. The air entered the cooling chamber from the bottom. A half-inch pipe is run out through ceiling to allow the specimen to rest on the bottom of the cooling chamber. A glass gauge would probably be





Photo-  
graph  
No.2



Photograph No.3





produced unequal cooling due to the air jet not expanding immediately to fill the 3-in. pipe. A basket of wire gauze was therefore slung inside the pipe to arrest the specimen about 8 inches from the bottom.

Temperatures were measured with a Keiser & Schmidt (Berlin) platinum rhodium couple pyrometer, with bare knuckle. The pyrometer ends were put thru the back wall of the furnace projecting about halfway into the muffled space.

Method.

A temperature range of 900°C. to 1300°C. was decided on as embracing all the possible desirable temperatures. Specimens of each of the steels were therefore treated at 920°C., 980°C., 1040°C., 1100°C., 1160°C., 1210°C., and 1280°C.

Twelve specimens were placed on the floor of the muffle at once and as the temperature rose to the first desired point it was held there for 10 minutes, then the air blast was turned on and one specimen of each steel was quickly seized and dropped into the cooling chamber. The temperature was then raised to the next designated point and held there the same length of time prior to quenching as before. The procedure was repeated until all these steels had been treated at all the designated temperatures.

The ends of the heat treated specimens not

produced the heat cooling the air and  
not necessarily immediately to the 1-1.4 gms.  
A basket of wire gauze was suspended in-  
side the air to prevent the solution about 6 in-  
ches from the bottom.  
Temperatures were measured with a Reissner  
Coulomb (Berlin) platinum resistance couple py-  
rometer, with bare wires. The pyrometer ends  
were not torn the face of the furnace, but  
rested about halfway into the muffle space.  
Notes.  
A temperature range of 0.01 to 1.00°C.  
was decided on as embracing all the possible  
feasible temperatures. Measurements of each of the  
series were therefore made at 0.00°C., 0.50°C.,  
1.00°C., 1.50°C., 2.00°C., 2.50°C., and 3.00°C.  
The muffle was cooled by the flow of  
the water at once and the water was kept at  
the first desired point it was held there for  
10 minutes, then the air blast was turned on and  
one specimen of each steel was rapidly cooled and  
dropped into the cooling chamber. The temperature  
was then raised to the next desired point and  
held there the same length of time prior to quench-  
ing the bottom. The measurements were repeated until  
all three series had been cooled at all the de-  
sired temperatures.  
The work of the present series was not

touched by the tongs were broken off at lengths of about one and a half to two and a half inches and these lengths were set aside for the metallographic work.

The atmosphere of the muffle was approximately neutral, because of the carbon lining, so that excessive oxidation of the specimens did not occur. This fact and the precautions observed are believed to have rendered the procedure of heat treatment described above free from material objection.

### Metallography

The heat treated specimens including pieces of each of the steels in the annealed condition, were polished, etched, and photographed under a fairly high magnification.

### Polishing

The polishing apparatus used was a Sauver vertical outfit. All polishing on the Sauver outfit is done on the flat side of plates mounted on a horizontal shaft. The plates include a grindstone and cast iron plates on which may be stretched canvass or soft buffing cloth.

The specimens were ground to a flat surface on the grindstone, or, in some cases, on a piece of emery cloth fastened to a flat plate in a high speed lathe. The flat surface was then finished, in turn, on wet emery paste, on canvas, wet tripoli paste on canvass and rouge on buffing cloth. The polishing was carried out until no scratches



were visible under 500 diameter magnification.

### Etching.

The following methods of etching were tried on the high speed steel.

Timed immersions from five to ten minutes in a 2% solution of picric acid and 95% alcohol, followed by quick washing and drying.

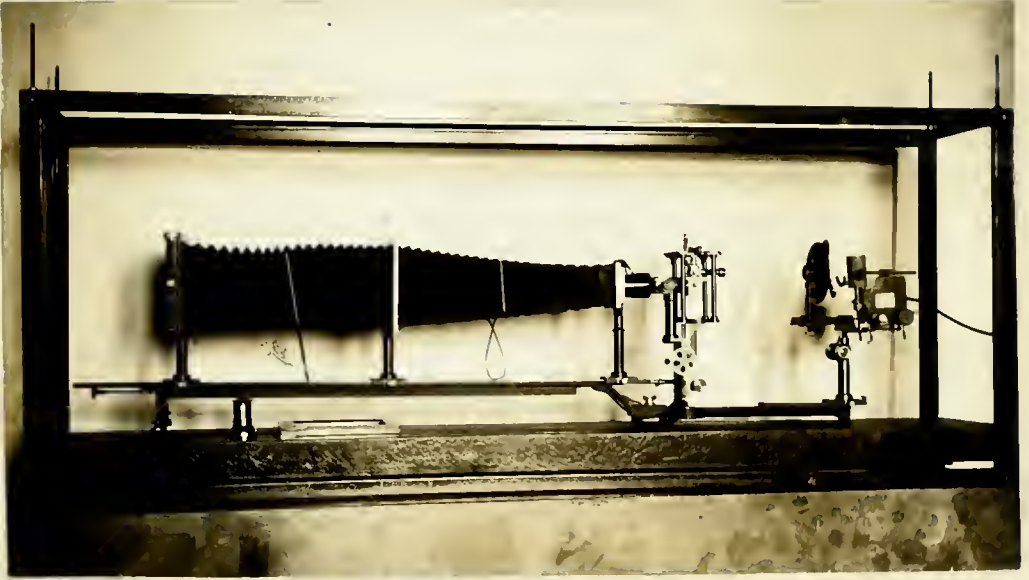
Timed immersions from five to ten minutes in the same picric acid solution followed by one second immersion in 10% nitric acid in water, followed by quick washing and drying.

Immersions of two to fifteen seconds in concentrated sulphurous acid followed by quick washings under running water dipping in alcohol and drying.

The picric acid etching gave fair results, but the sulphurous acid method was far shorter and equally satisfactory. The etching method adopted consisted in dipping the specimen in sulphurous acid, washing in running water, dipping in alcohol and drying with soft cotton. The specimen was then examined under the magnification to be used later in photographing and if more etching seemed desirable, a second or a third short treatment with sulphurous acid was employed. When the surface oxidized badly as a result of the etching, the oxide film was removed by rubbing on electro-silicon on a chamois skin.







Photograph No.4





### Microphotography.

A large metallurgical camera manufactured by E. Leitz, of Wetzlar, was available for the photographic work. Photograph No. 4 shows this camera, which includes an arc light, object stage, microscope and camera bellows, placed on a bed which is slung on springs in such a manner that any slight motion communicated to the apparatus does not disturb the relative position of the parts. An equipment of lenses allows a great variety of work with this camera. Its operation requires close familiarity with the various adjustments, as these are many and must be correct to obtain good results.

All the photographs were taken at a magnification of 500 diameters. The etched specimen was placed on the object stage, brought into sharp focus, and exposed for two and a quarter to two and a half minutes (depending on the surface) with a small opening in the diaphragm regulating the light supplied and a green glass screen interposed as a ray filter.

Five inch by seven inch photographic plates (Imperial S. S., supplied by Gennert) were used. These were developed with metol solution and prints were taken on hard finish special contrasty paper.

The prints contained are included in the following pages.



Poldi -- Annealed



Poldi -- Treated at 9200C



Amesbury

--

1904

Treated at 2000

--

1904



Poldi

--

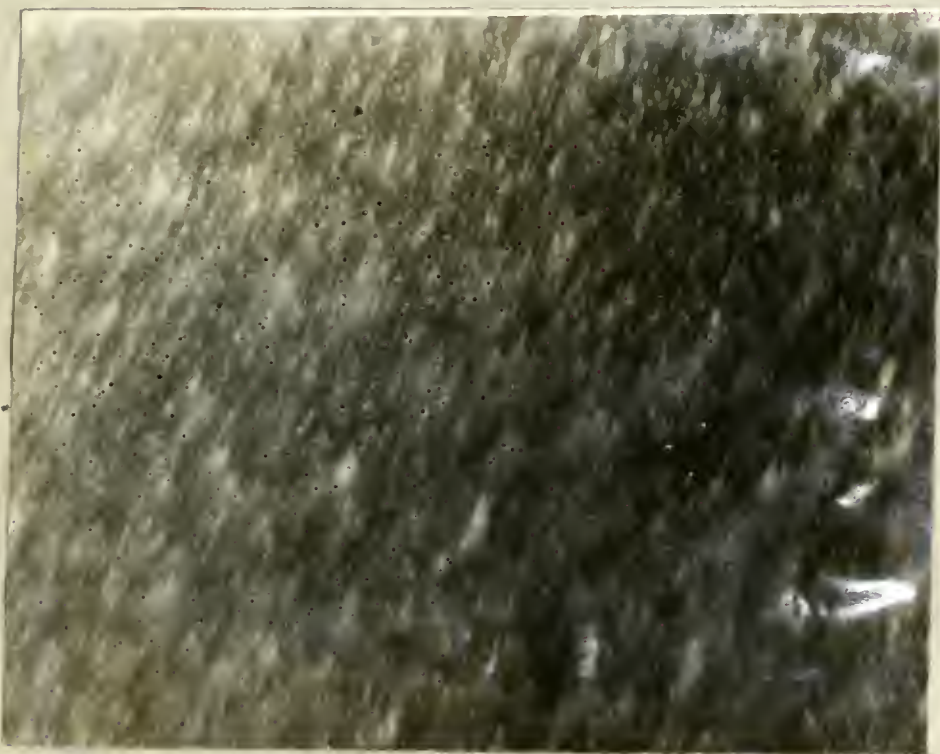
Treated at 980°C



Poldi

--

Treated at 1040°C





10/10/10

10/10/10

10/10/10

10/10/10

10/10/10

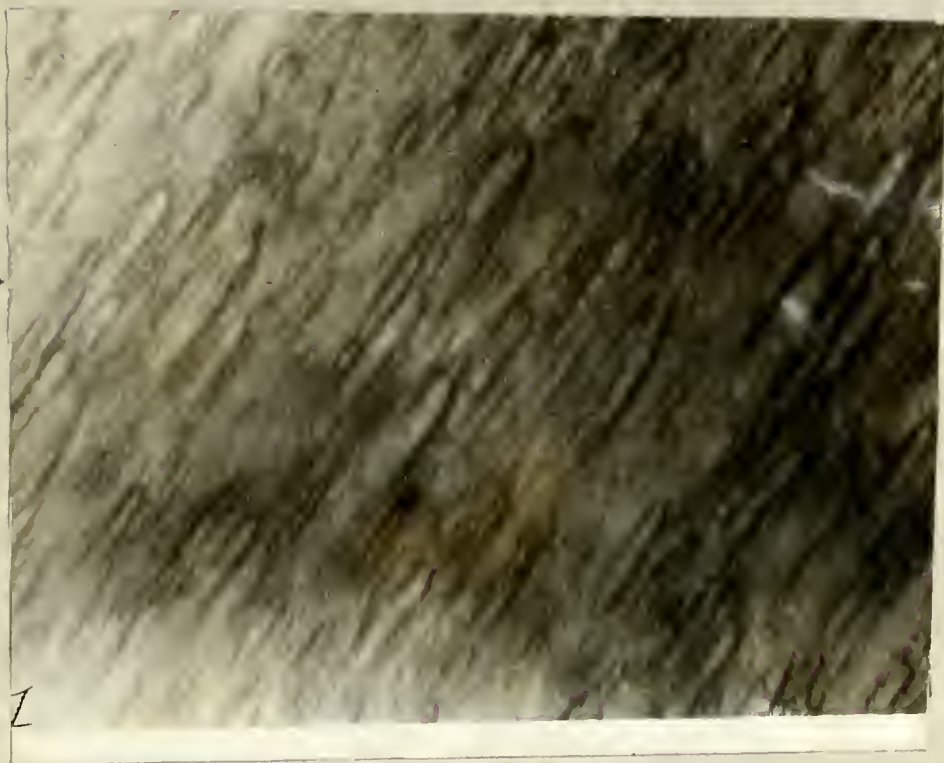
10/10/10

10/10/10

Poldi

--

Treated at 1100°C



Poldi

Treated at 1160°C



Treated at 1100°C

--

12.10.4



12.10.4

Treated at 1100°C

12.10.4

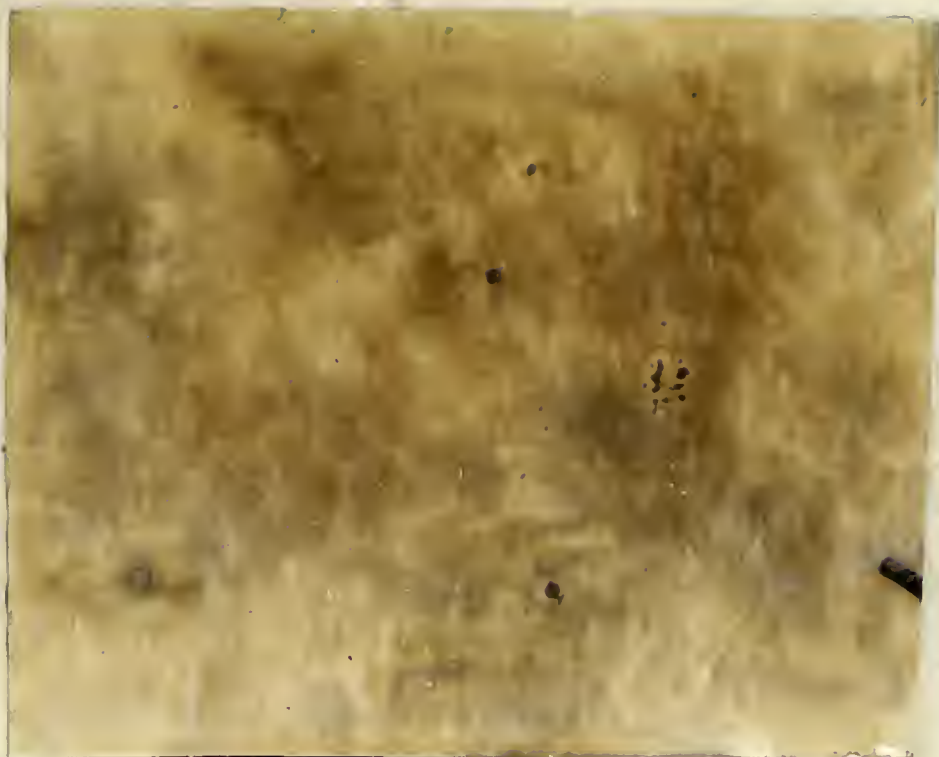
12.10.4



Poldi

--

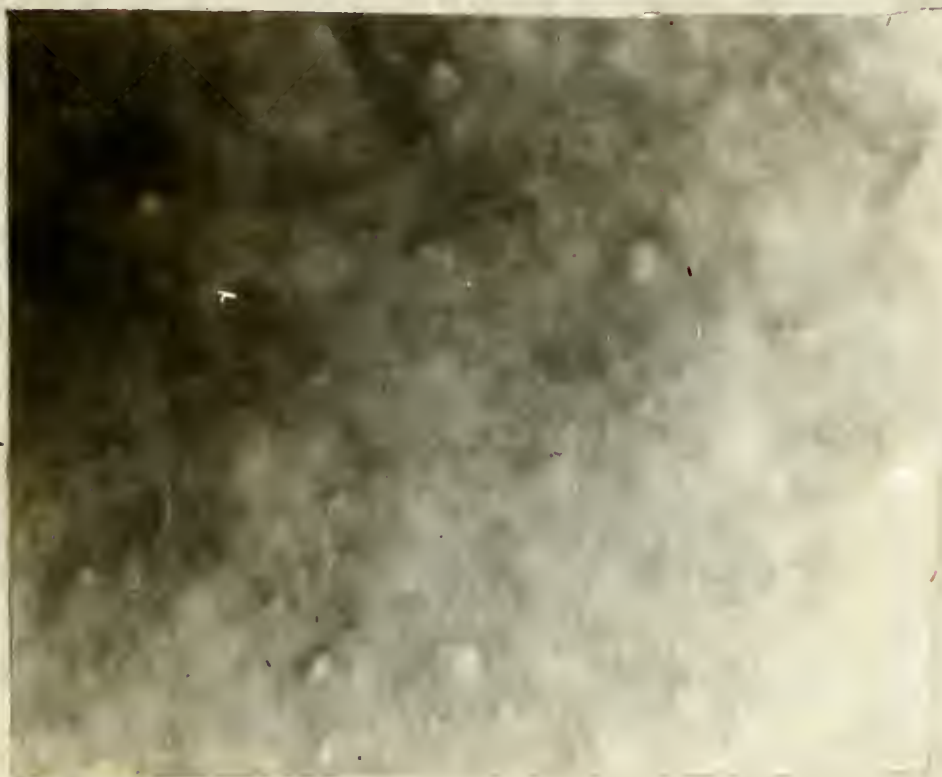
Treated at 1210°C



Poldi

--

Treated at 1280°C



Treated at 12500

Point

Treated at 12500

Point



Arc

--

Annealed



Arc

--

Treated at 920°C





Annexed

--

are

Tested at 200°

--

are

Arc -- Treated at 980°C



Arc -- Treated at 1024°C



Treated at 280°C

--

27A

Dec 4 1942

Treated at 1034°C

--

27A

Dec 10 1942

Arc

--

Treated at 1100°C



Arc

--

Treated at 1160°C



100000 to 100000 -- on

100000 to 100000 -- on



Arc -- Treated at 1210°C



Arc -- Treated at 1220°C





1911

1912

Bethlehem

--

Annealed



Bethlehem

--

Treated at 920°C



1955-1957

56175

20 21

$$n^2 \log n + n \log n$$

1891

□ □

Bethlehem -- Treated at 980°C



Bethlehem -- Treated at 1040°C



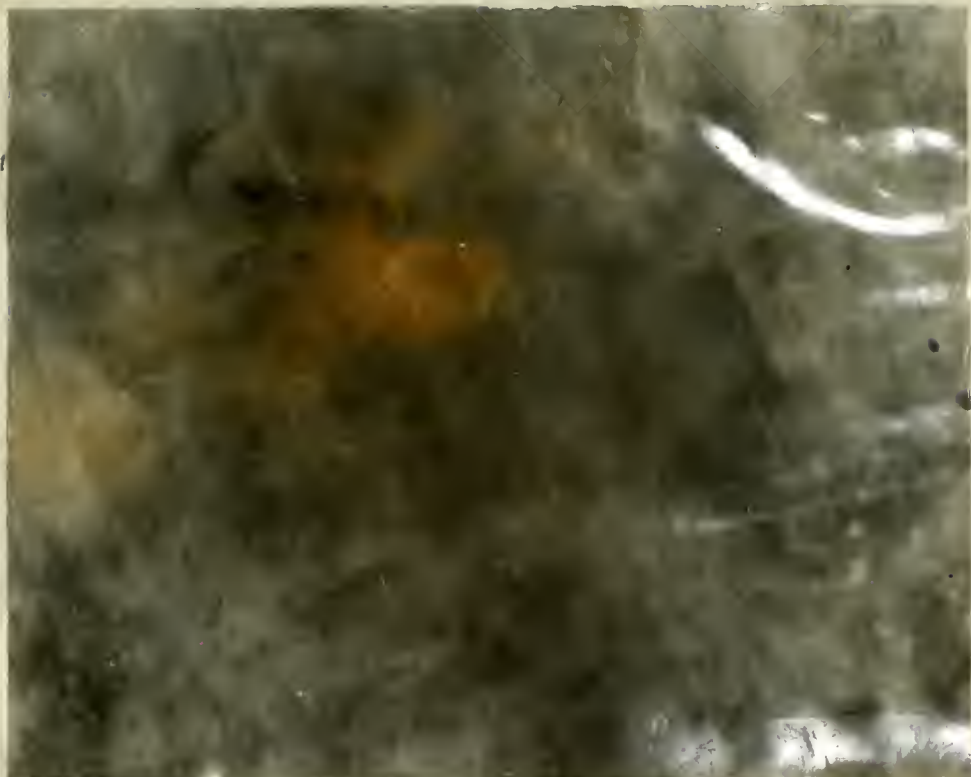




Bethlehem

--

Treated at 1100°C



Bethlehem

--

Treated at 1160°C





Retained -- Treated at 1100°

Total -- Treated at 1100°

Bethlehem

--

Treated at 1210°C



Bethlehem

--

Treated at 1280°C



Water Resources

Water Resources

Water Resources

Novo

--

Annealed



Novo

--

Treated at 9200C







Novo

--

Treated at 980°C



Novo

--

Treated at 1040°C





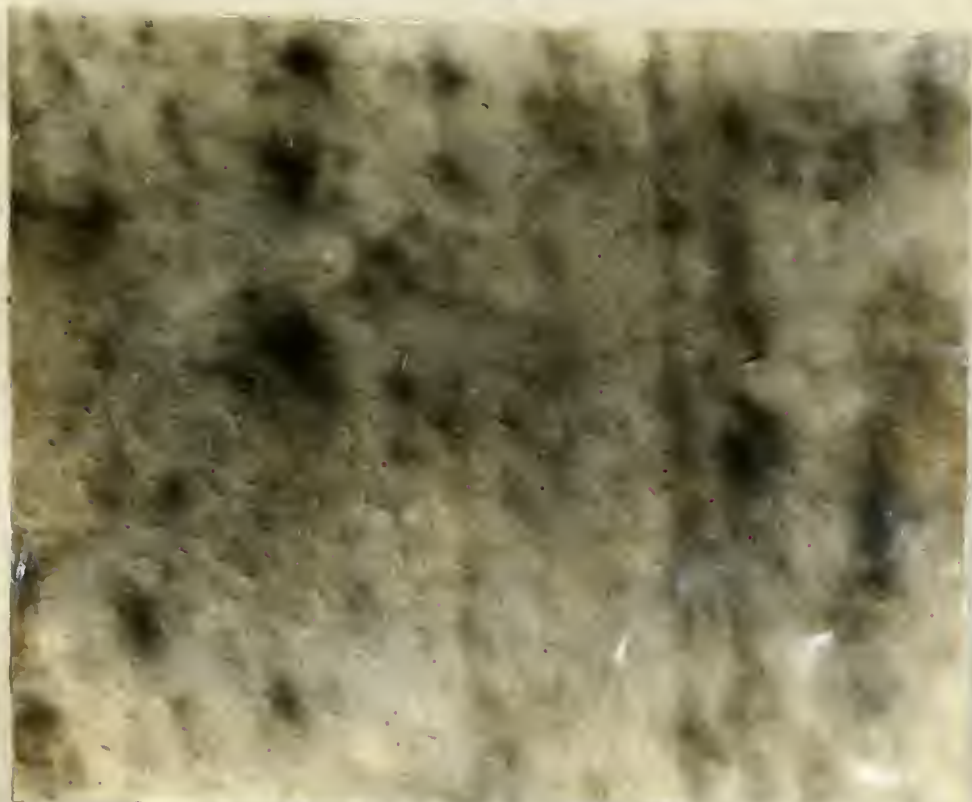
Handwritten notes in the first section.

Handwritten notes in the second section.

Novo -- Treated at 1100°C



Novo -- Treated at 1160°C



Printed at 1100°

over

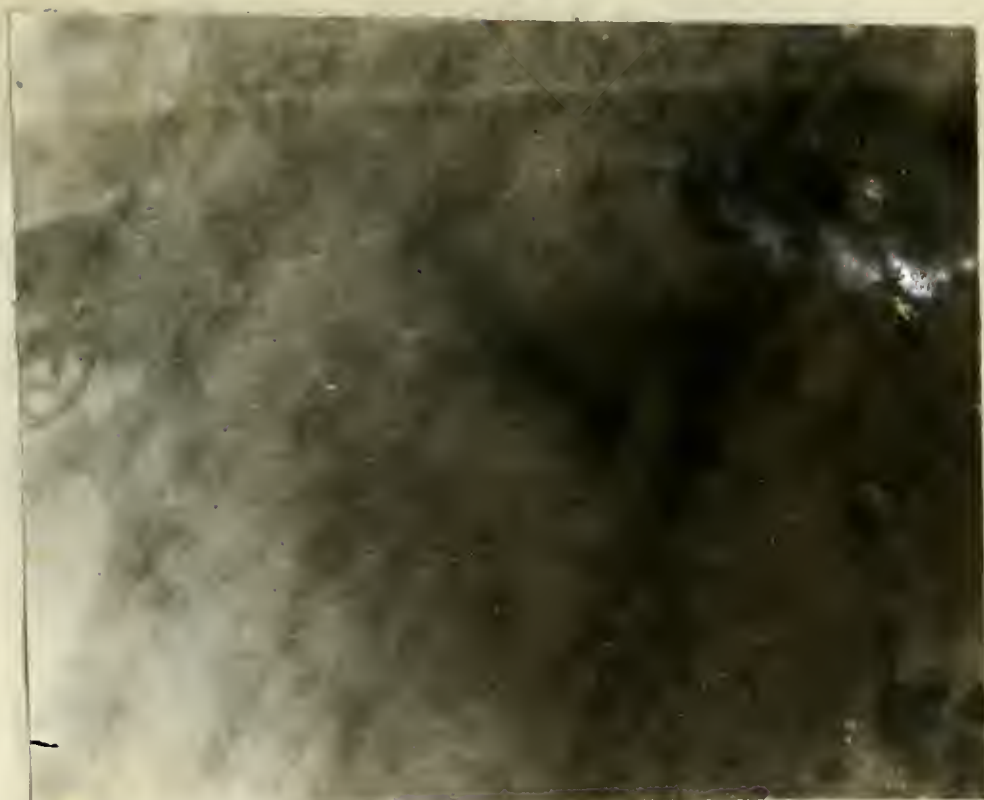
Printed at 1100°

over

Novo

--

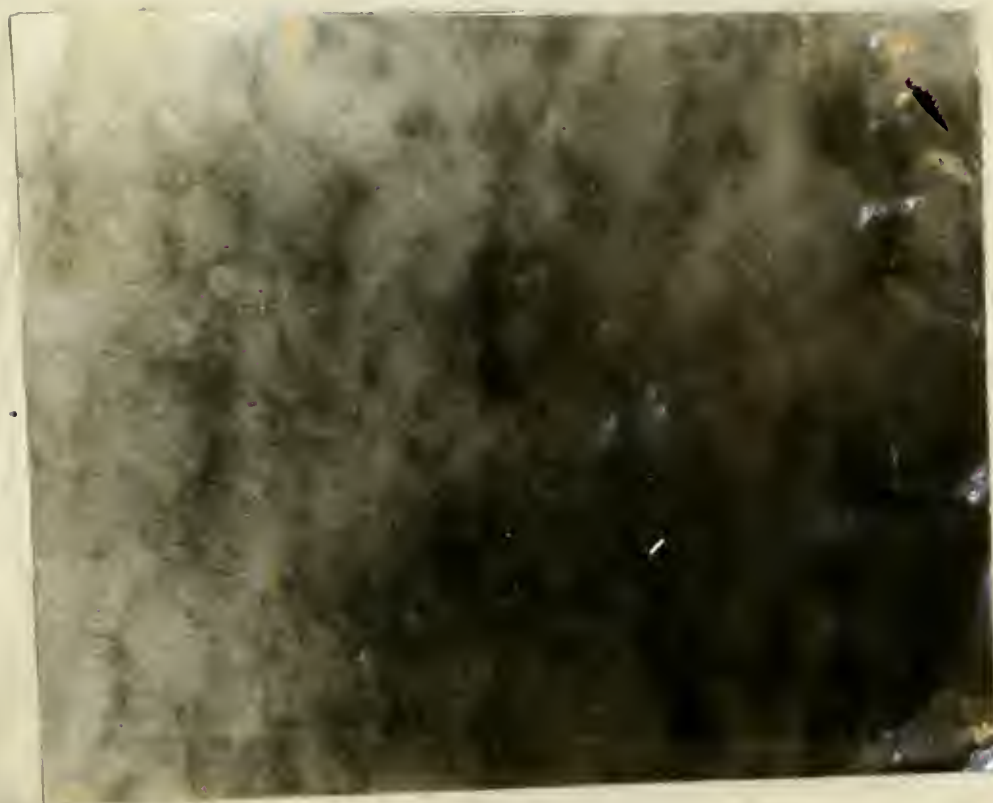
Treated at 1210°C



Novo

--

Treated at 1280°C



10/10/10



Rekord

--

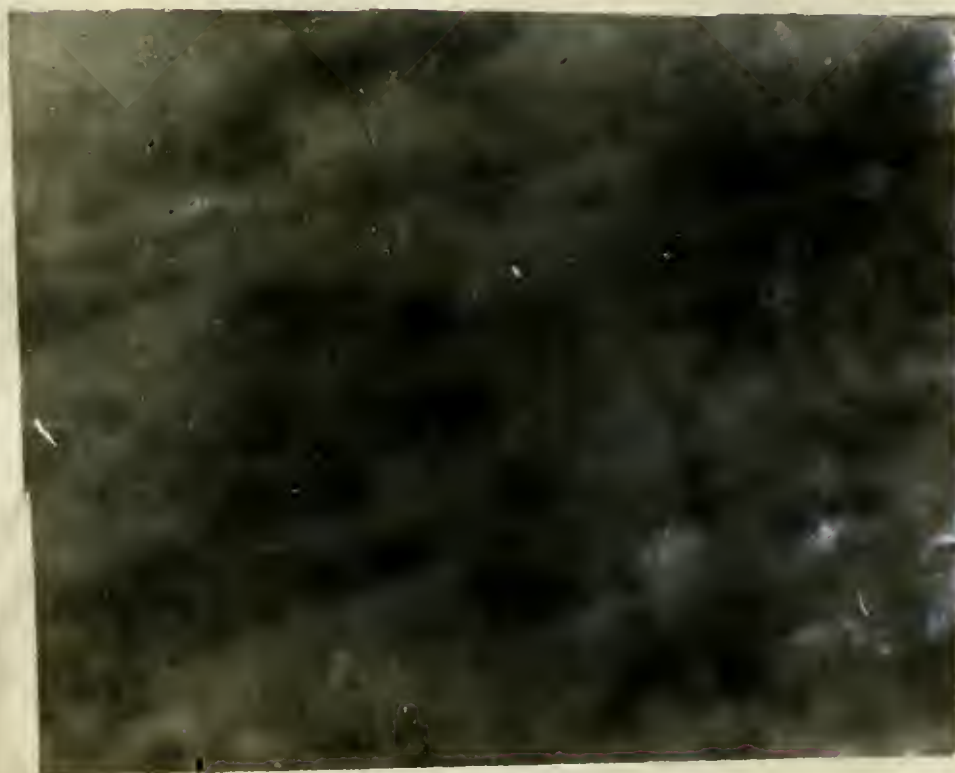
Annealed



Rekord

--

Treated at 920°C





John E. Smith

1870

John E. Smith

Rekord

--

Treated at 980°C



Rekord

--

Treated at 1040°C



Printed at 8000

--

Printed

1898

Printed at 1000

--

Printed

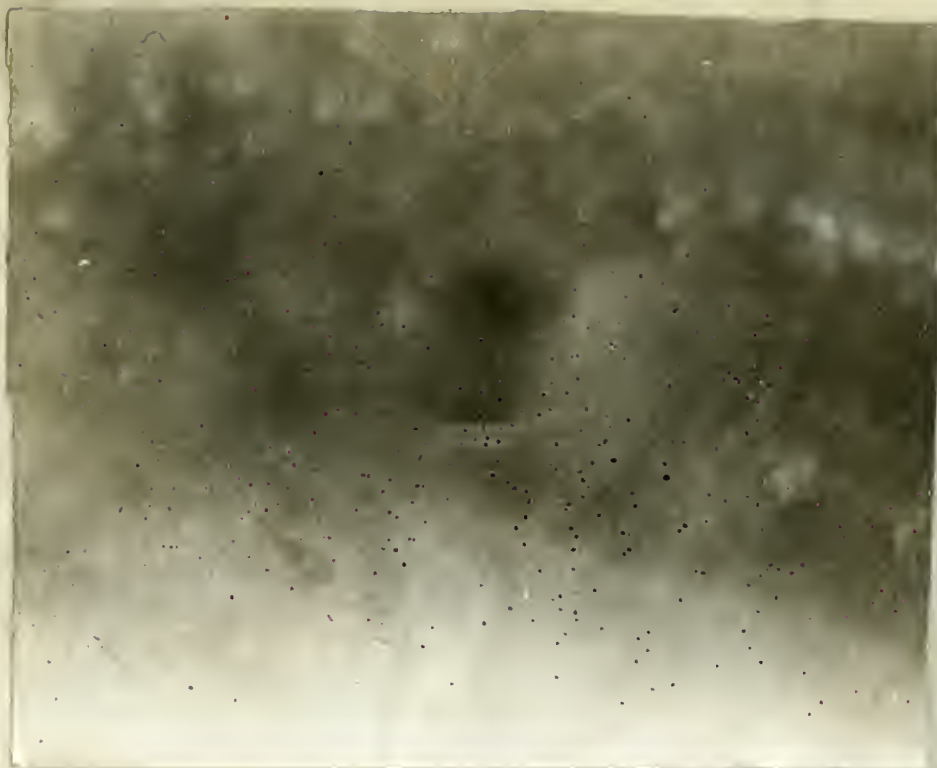
1898

1898

Rekprd

--

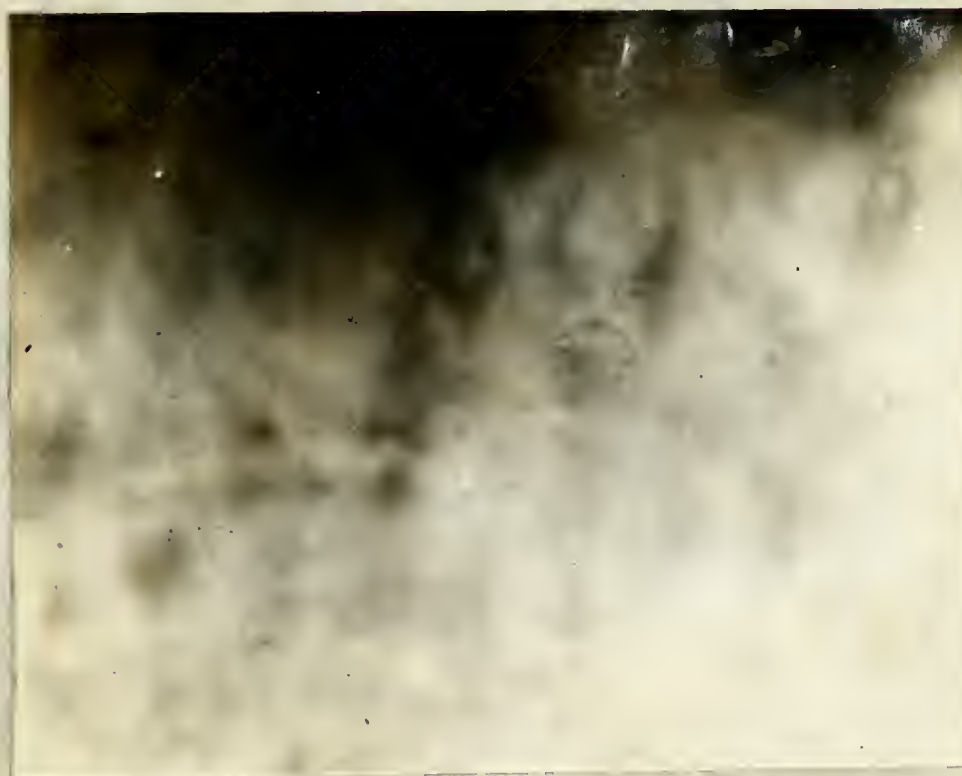
Treated at 1100°C



Rekord

--

Treated at 1160°C



1000

1000

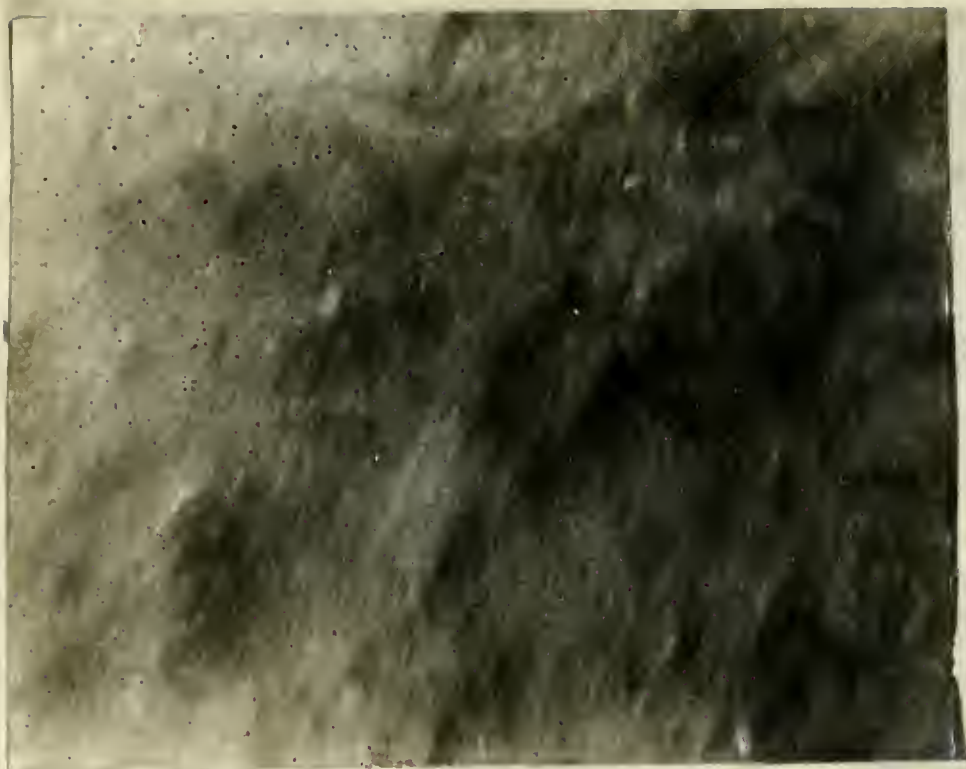
1000



Rekord

--

Treated at 1210°C



Rekord

--

Treated at 1280°C





Record

Traded at 1000

1000

1000

Record

Traded at 1000

1000

Blue Chip

--

Annealed



Blue Chip

--

Treated at 920°C



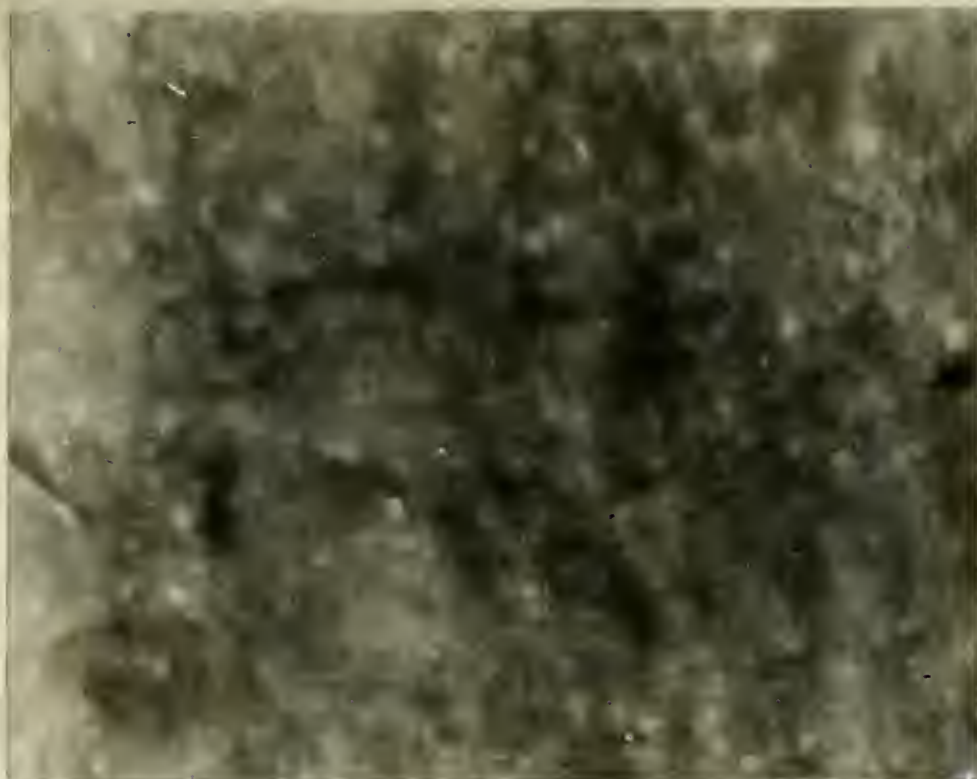
Handwritten notes and sketches, including a large, stylized drawing of a person or figure, possibly a deity or a historical figure, with long, flowing hair or robes. The drawing is done in a simple, sketchy style with some shading.

Handwritten notes and sketches, including a large, stylized drawing of a person or figure, possibly a deity or a historical figure, with long, flowing hair or robes. The drawing is done in a simple, sketchy style with some shading.

Blue Chip

--

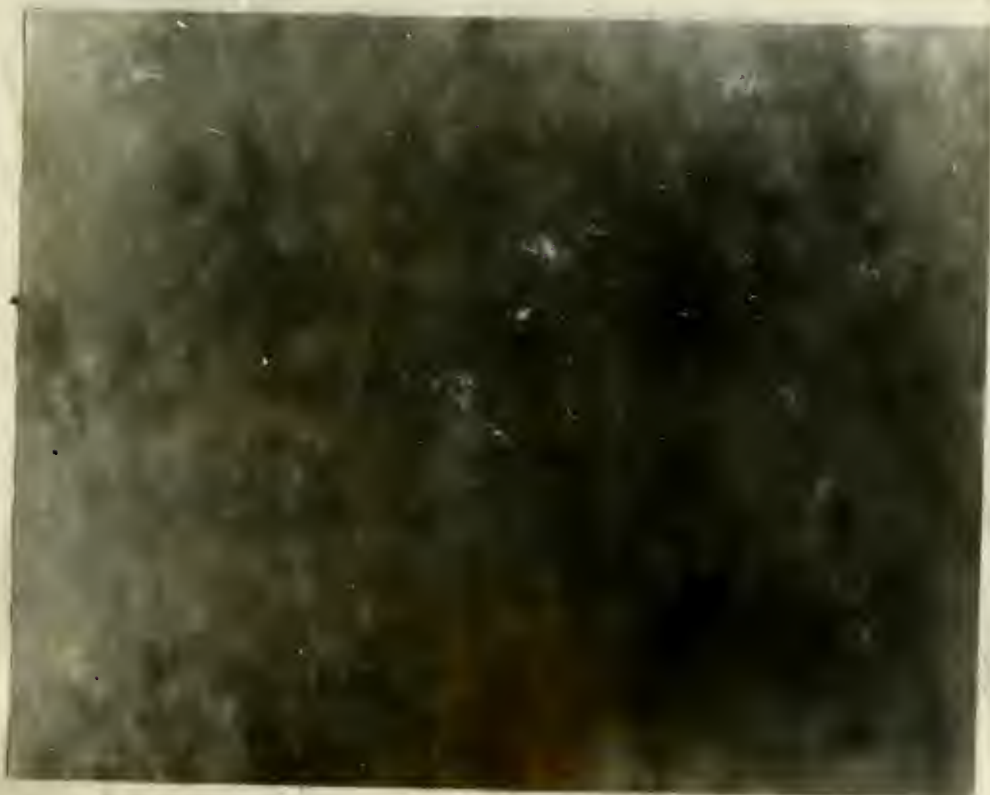
Treated at 980°C



Blue Chip

--

Treated at 1040°C



Treated at 30°C

Blue chip

C. C. 40

Treated at 104°C

Blue chip

C. C. 40



Blue Chip

--

Treated at 1100°C



Blue Chip

--

Treated at 1160°C



Treated at 11500

Blue Chip

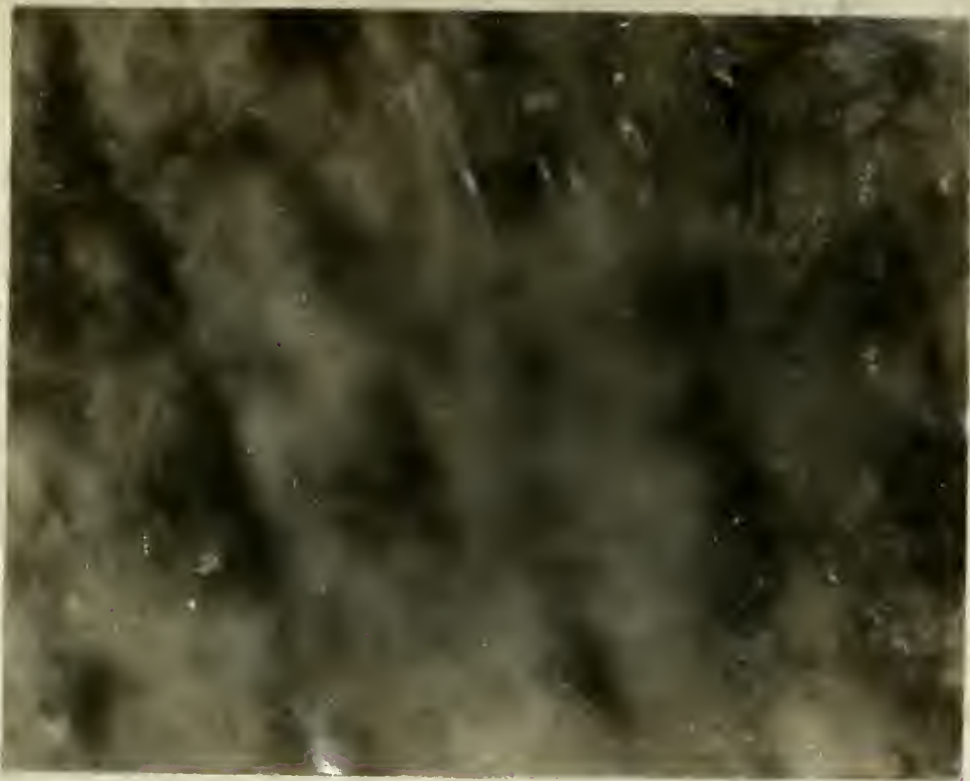
Treated at 11500

Blue Chip

Blue Chip

--

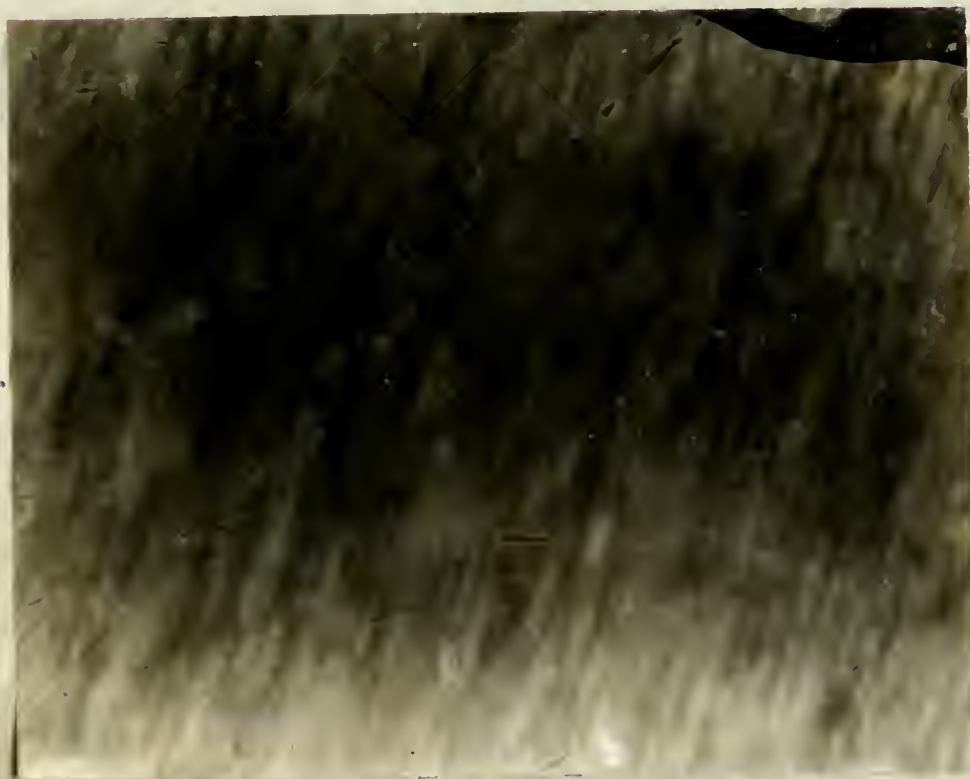
Treated at 1210°C



Blue Chip

--

Treated at 1280°C



Blue Chip -- Treated at 1960°C

8-2-10

Blue Chip -- Treated at 1960°C

8-2-10



### Interpretation of Results and Conclusions.

To give the best results as a cutting tool, high speed steel must possess, in addition to hardness, enough toughness and resilience to withstand a certain degree of shock. In the present case where the attempt is to determine toughness, or brittleness and hardness, from microphotographs showing the structure of the steel, established standards are necessary to permit definite statements. On carbon steels and irons, enough work has been done to make it possible to pass accurate judgment on a specimen by obtaining a good microphotograph of it. In the case of the chrome tungsten steels, this does not hold true. Work has been done but it is not sufficient.

The prints on the preceding pages can be commented on only by inference from what is known definitely regarding such materials as carbon steels and from a small amount of direct knowledge concerning high speed steel.

It is probable that in the high speed steels as elsewhere, coarse structure is an indication of a tendency toward brittleness. This is borne out by the observation that those specimens showing particularly coarse structures broke readily under the hammer. Conversely, it is probably true that fine homogenous structures indicate that the specimen will stand shock. Also a marked cellular structure in a chrome tungsten steel is interpreted as indicating the presence of a double





carbide of tungsten which lends hardness to the specimen.

Referring to the microphotographic prints of Roldi, it is seen that the annealed specimen shows fairly large crystals, in a striated and rather non-homogenous field. At 920 degrees, this original structure has broken up, resulting in smaller crystals set in a fairly homogenous field. From 920 degrees to 1040 degrees, there seems to be a steady growth in the size of the crystals and an increase in the degree of separation between them - the compound crystals are segregated more distinctly from the ferrite matrix. At 1100 degrees a banded homogenous structure rather coarse crystalline, appears and at 1160 degrees the band markings have almost disappeared, the crystals have grown smaller and cellular grains appear. At 1210 degrees, grain markings are disappearing and the crystals have elongated, while at 1280 degrees, a coarse non-homogenous structure appears.

Between 1100 degrees and 1160 degrees we pass from a typically banded structure to a homogenous close cellular grain structure. On the assumption that the cellular structure indicates hardness ( see above ) the best temperature for this steel may be supposed to be between 1100 and 1160 degrees, the cells having grown somewhat larger than is supposed desirable at the latter temperature.

The annealed specimen of Arc shows a non-homogenous field of moderate sized crystals, and a striated

... of the ... to the ...  
... to the ...  
... it is ...  
... in a ...  
... at 1200 degrees, ...  
... resulting in ...  
... from the ...  
... to be a ...  
... and an increase in ...  
... the composition ...  
... from the ...  
... composition ...  
... and at 1100 degrees ...  
... up ...  
... 1100 degrees ...  
... appearing and ...  
... 1200 degrees, ...  
... Between 1100 ...  
... a typically ...  
... grain structure ...  
... structure ...  
... for this ...  
... 1100 and 1150 degrees, ...  
... somewhat larger than ...  
... temperatures.  
... The ...  
... 1100 ...

9-0

structure, which has not completely broken up at 920 degrees, though the crystals have decreased in size. At 980 degrees a rearrangement into coarser crystals appears, showing non-homogenous areas and a remnant of the striated structure. At 1040 degrees, smaller crystals appear in a more homogenous field and at 1100 degrees the field is quite homogenous. At 1160 degrees large cellular grains are marked in a still homogenous field from which crystalline structure has largely disappeared. A rearrangement is noted at 1210 degrees, resulting in a field showing non-homogenous areas and a loss of the cellular markings, which appear once more as the outline of large sized grains in an extremely non-homogenous field at 1280 degrees.

From the trend of this series of prints, it seems likely that at slightly above 1100 degrees, a structure possessing the characteristics of homogeneity with fine crystalline structure and yet marked by close cellular grains would be obtained. The best temperature, in this case, seems therefore, in that vicinity.

Bethlehem seems to lose the banded structure of the annealed specimen when treated at 920 degrees, but retains the original non-homogeneity and shows large crystals. At 980 degrees, the crystals are rearranged and segregated to sharper separation from the ferrite matrix and a return to the banded structure is







noted, and at 1040 degrees the crystals have elongated and grown in size, but the field is fairly uniform. From 1040 to 1210, a progressive decrease in the size of the crystals is noted with the result that at 1210 degrees, we have a very fine homogenous crystalline structure. At 1280 degrees, a coarse cellular structure is plainly shown - probably indicating a very hard, yet brittle specimen.

The fine homogenous field at 1210 degrees and the large grains at 1280 degrees lead to the opinion that slightly above 1210 degrees may be designated as the temperature that would produce the best results with Bethlehem.

The slightly banded non-homogenous field shown by annealed iron does not show complete rearrangement until 980 degrees is reached. Here we have moderate sized crystals in a homogenous field. At 1040 degrees there is noted a second complete rearrangement resulting in an unique field showing non-homogenous areas containing coarse crystals markedly segregated. At 1100 degrees, the field is homogenous and contains moderate sized crystals, while at 1160 degrees, it is again non-homogenous and shows segregation, though the structure is not so coarse as at 1040 degrees. 1210 degrees presents a fairly homogenous field with smaller structure supposed to indicate hardness. The photographs here are not particularly decisive. Probably a point between 1210 degrees and 1280 degrees could be designated as the



best temperature, but 1100 degrees also shows a good structure.

The annealed specimen of nokord shows fairly large crystals in a rather homogenous field. 920 degrees brings a rearrangement resulting in a field containing non-homogenous areas, which feature persists at 980 degrees, though the crystals are fairly small here. At 1040 degrees the field is more homogenous but shows fairly marked segregation and large crystals, which, at 1100 degrees have become small and are close set into a homogenous structure. As far as can be judged from the print the structure at 1160 degrees resembles that at 1100 degrees. At 1210 degrees there is a coarse field with non-homogenous areas which features are more emphasized at 1280 degrees.

It is remarked that none of the prints of this series shows distinct granular structure. The best structures seem to occur at 1100 to 1160 degrees.

Heating to 920 degrees changes the banded structure of annealed Blue Chip to a homogenous structure of fairly large crystals distributed through a matrix. From 920 degrees to 1040 degrees the most noticeable change is a loss of homogeneity. From what can be learned from the print there is no marked segregation at 1100 degrees but at 1160 degrees large cellular grains appear and the segregation has occurred with a loss of homogeneity. At 1210 degrees the cellular markings are not seen, but at

11

structure.

The analysis of the structure of the

crystals in a rather homogeneous field.

shows a certain amount of variation in a field of

non-homogeneous fields, which is shown in the

though the crystals are fairly small here.

degrees the field is more homogeneous but some fairly

marked segregation and large crystals, which at 1100

degrees have become small and are close to a

homogeneous structure. At this point the

print the structure at 1100 degrees is

1100 degrees. At 1100 degrees there is a

with non-homogeneous structure which is

observed at 1100 degrees.

It is remarked that some of the points of

shows distinct structural changes. The

seem to occur at 1100 degrees.

Heating to 1100 degrees changes the

of a material into a homogeneous structure

large crystals distributed through a matrix.

degrees to 1100 degrees the most noticeable change is

loss of homogeneity. The material can be

print there is a marked segregation at 1100 degrees

at 1100 degrees large crystals are

segregation has become with a loss of

1100 degrees the material is



1280 degrees there is a slight reappearance of these markings.

Some desirable characteristics, as judged from the photos, appear when Blue Chip is heated to 1100 degrees, while it is apparently hard at 1160 degrees, the best temperature probably lying between these two points.

While sufficient work has not been done to permit of any definite statements, it is noticed that a change of 60 degrees C. in the temperature to which a chrome tungsten high speed is heated prior to quenching results in a marked structure change, in most cases. It would seem, therefore, that careful investigation should reveal a temperature, unique for each steel, that should be closely adhered to, to obtain the best results. It seems worth while to prove or disprove, by careful study, the idea that a temperature for heating prior to quenching can be found which will give much better results than a temperature only slightly above or below.

It is believed by the authors that definite conclusions could be arrived at by studying a large number of specimens by making a number of physical tests - tensile, transverse, cutting and hardness - together with the taking of microphotographs. In this way, sufficient general knowledge might be acquired to enable passing accurate judgment on other specimens solely by examining the microphotographs. The indications obtained in the work done, show distinct possibilities of





obtaining good results from a careful, thorough and lengthy investigation of the problem.

It will be noted that there is an indication that the best temperature for heat treating is highest in the steels containing the largest per cent of tungsten. This may or may not be coincidence.



# TABLE I

## GUIDE TO MARKED SPECIMENS

TEMPERATURE °C.	ANN- SALED	920	980	1040	1100	1160	1210	1280
NUMBERS								
REKORD (SWOSEN)	0-0	0-1	0-2	0-8	0-4	0-5	0-7	0-6
BLUECHIP	1-0	1-1	1-2	1-8	1-4	1-5	1--	1-6
ARC (JESSOP'S)	2-0	2-1	2-8	2-5	2-4	2--	2-7	2-2
NOVO	4-0	4-1	4-2	4-8	4-4	4-5	4-7	4-6
BETHLE- HEM	5-0	5-1	5-2	5-8	5-4	5-5	5-7	5-6
POLDO	8-0	8-1	8-2	8-8	8-4	8-5	8-7	8-6

N. B. To read number on specimen on one plate read the number nearest the edge first. Thus  is specimen 1-2 in Table I and  is

plate 1-2 in Table II.  
(Look at plate number with gl. line. Toward the eye.)

Where labels have been placed on specimen (because of difficulty in reading the punch marks) read as indicated from left to right.

Tables I and II are not in exact correspondence, but both are correct.

# TABLE II

## GUIDE TO MARKED PLATES

TEMPERATURE °C.	ANN- SALED	020	980	1040	1100	1160	1210	1280
NUMBERS								
REKORD	0-7	8-2	0-1	0-2	8-1	0-5	0-4	0-6
BLUECHIP	1-0	1-1	1-2	1-8	1-4	1-5	1--	1-6
ARC	2-0	2-1	2-8	2-5	2-4	2--	2-7	2-2
NOVO	4-0	4-1	4-2	4-8	4-4	4-5	4-7	4-6
BETH.	5-0	5-1	5-2	5-8	5-4	5-5	5-7	5-6
POLDO	8-0	8-8	8-5	X	8-4	0-0	0-8	8-6

